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■

1975 AND SUBSEQUENT MODEL YEAR VEHICLES

Protection of Emission Control Systems

ENVIRONMENTAL PROTECTION AGENCY

[40 CFR Part 80]

[FRL 275-2]

FUELS AND FUEL ADDITIVES

Protection of Emission Control Systems; 1975 and Subsequent Model Year Vehicles

The Environmental Protection Agency promulgated regulations on January 10, 1973, that provide for the general availability of one grade of unleaded gasoline by July 1, 1974 (38 FR 1254). The regulations as amended[1] also prohibit the introduction of leaded gasoline into any motor vehicle which is labeled "unleaded gasoline only" or which is equipped with a gasoline tank filler inlet designed for the introduction of unleaded gasoline. The purpose of these regulations is to protect the emission control systems that will be installed on 1975 and subsequent model year vehicles. The regulations also establish labeling and equipment requirements for pumps for the sale of unleaded gasoline.

On July 3, 1974, EPA promulgated laboratory test procedures to measure the lead and phosphorus content of gasoline. These test methods will be utilized to test samples of gasoline represented as unleaded to determine whether the fuel has been contaminated. If these tests indicate that the gasoline contains excessive lead or phosphorus, EPA will initiate proceedings to determine which party in the chain of distribution caused the violation. Civil penalties will subsequently be assessed upon those ultimately held responsible.

The necessity for monitoring unleaded gasoline results from the degradation of certain emission control devices when exposed to gasoline contaminated with lead or phosphorus. In particular, catalytic converters which will be installed on 65 percent to 80 percent of 1975 model year vehicles are sensitive to very small quantities of these elements. The catalyst oxidizes the exhaust gas pollutants of carbon monoxide and hydrocarbons to carbon dioxide and water. Lead and phosphorus form compounds which coat the active materials in the converter and create a barrier which the exhaust gases must penetrate before oxidation can occur. As this barrier increases, the efficiency of the catalyst diminishes and eventually the pollutant emissions emanating from the vehicle exceed federal standards. For this reason, unleaded gasoline containing no more than 0.05 gram of lead per gallon and 0.005 gram of phosphorus per gallon will be required for 1975 and later model year vehicles equipped with catalytic converters.

Since the deleterious effect of excess lead and phosphorus on catalyst efficiency is cumulative, it is imperative that every effort be made to avoid the contamination of gasoline represented as unleaded and, when it does occur, to prevent its introduction into catalyst-equipped cars. Thus, when contaminated unleaded gasoline is discovered through the EPA enforcement activities, it is not sufficient to merely assess penalties against those parties liable for causing the violation. In addition, EPA must be able to immediately stop the sale of contaminated unleaded gasoline as soon as it becomes apparent that contamination exists.

Section 80.22(a) prohibits retailers from selling, dispensing, or offering for sale contaminated gasoline as unleaded and from causing or allowing the introduction of leaded gasoline into a vehicle labeled "unleaded gasoline only." The purpose of the regulations proposed below is to supplement § 80.22(a) by providing a procedure that will more readily permit the enforcement of the prohibitions against the introduction of contaminated gasoline into a vehicle requiring unleaded fuel.

In order to facilitate enforcement of § 80.22(a), EPA has been working in conjunction with the American Society of Testing and Materials (ASTM) to develop a standardized field test method for measuring the lead content in gasoline dispensed from unleaded pumps. As a result, ASTM has developed a test which may be utilized by EPA fuel inspectors in the field to determine with a 99 percent or greater level of confidence whether the amount of lead in a sample of gasoline exceeds the federal maximum. EPA now proposes to adopt the ASTM test method for a field determination of lead in gasoline represented to be unleaded (hereafter referred to as the "field test").

The field test will usually be performed at the retail outlet where gasoline is stored and dispensed. The test results will be the inspector's basis for notifying the retailer that the fuel samples are in apparent violation of the federal standard of 0.05 gram of lead per gallon. Then, under the proposed regulations, sale of any gasoline from a pump served by the storage tank containing the contaminated fuel will be prohibited. By prohibiting the sale of this gasoline from an unleaded pump to any vehicle, both those requiring unleaded fuel as well as those which can operate on leaded product, an EPA inspector can verify by a meter reading that this contaminated gasoline was not dispensed through the unleaded pumps after the retailer was notified. Pumps served by the storage tank containing the contaminated gasoline would also be posted with a notice of contamination under these proposed regulations. This notice would remain posted until such time as the contamination is cured or disposal is made of the gasoline.

Prohibition of sales on the basis of an on-site field test is required in order to preclude catalyst deactivation during the period of time between detection of the apparent violation and confirmation by the laboratory test. In addition to constituting a violation under the present § 80.22(a), the regulation proposed below will add an additional paragraph which will make it a separate violation to sell contaminated gasoline from a pump subject to a notice of contamination after the retailer has been notified of the contamination.

At this time, no field test to determine the amount of phosphorus in gasoline is proposed for adoption. However, EPA will take immediate action to notify retailers whose gasoline has been found to be contaminated on the basis of the phosphorus laboratory test as well as from a lead laboratory test whether or not the field test has been performed on the sample.

The statutory authority for the prohibition discussed above is contained in section 211(c)(1)(B) of the Clean Air Act which states, in part, that the Administrator may "by regulation, control or prohibit the manufacture, introduction into commerce, offering for sale, or sale of any fuel or fuel additive for use in a motor vehicle or motor vehicle engine * * * if emission products of such fuel or fuel additive will impair to a significant degree the performance of any emission control device or system which is in general use * * *."

The sale or introduction of gasoline represented to be unleaded and contaminated with lead or phosphorus into a motor vehicle equipped with a catalyst constitutes a circumstance in which significant impairment of the catalyst will occur. Control of unleaded gasoline for this purpose was explicitly approved by the Senate Public Works Committee when it reported the Clean Air Act Amendment to the Senate. As stated in Senate Report No. 91-1196 of September 17, 1970:

> Another reason for prohibiting or controlling the sale of a fuel involves its effect on an emission control device or system. The most obvious example that came to light in hearings is the effect of leaded gasoline on a catalytic muffler. Such mufflers have been proposed to control emissions of hydrocarbons and carbon monoxide from vehicle engines. The effect of lead on catalysts can reduce the effectiveness of such mufflers by up to 90 percent. Since the use of catalytic mufflers may be essential for compliance with standards established under section 202, the Committee has adopted language permitting the Secretary to control fuels in order to facilitate the use of emission control systems.

The field test proposed by these regulations will be contained in detail in Appendix C to Subpart A, 40 CFR Part 80, Regulation of Fuels and Fuel Additives. A Subpart C is proposed covering procedures in case of contamination, as follows:

(a) A sample of gasoline from a pump dispensing gasoline represented as unleaded will be collected by the EPA inspector.

(b) The sample will usually be tested for lead content in a mobile laboratory or its equivalent in accordance with the field test described in Appendix C. A measurement by that procedure of 0.07 gram per gallon or above for one sample, for example, indicates at least a 99 percent level of confidence that the gasoline contains lead additives in excess of 0.05 gram per gallon.

[1] 38 FR 28301, October 12, 1973; 39 FR 13174, April 11, 1974; 39 FR 16123, May 7, 1974; 39 FR 16137, May 7, 1974.

(c) If the field test indicates with an adequate level of confidence that the gasoline contains lead in excess of the federal standards, contamination will be indicated. In order to assure test accuracy, it is contemplated that tests indicating contamination will be interspersed at appropriate intervals with the same test conducted on a standard fuel sample which contains a predetermined quantity of lead. If the results of the test of the standard correspond with the known amount of lead in the sample, the inspector will be certain that the testing equipment is properly calibrated, the reagents are in good working order, and he is following proper testing procedures. A second fuel sample may be taken from the unleaded pump and tested as described in (b) to increase EPA's confidence in the test results.

(d) The retailer or his employee or agent will then be notified of the contamination. This written notification will be signed by the Regional Administrator or his delegate and will inform the retailer: (i) That further sale of gasoline as leaded or unleaded from all pumps serviced by the same storage tank which serviced the pump from which the contaminated fuel sample was taken is prohibited by regulation; (ii) that the contaminated gasoline must be disposed of or otherwise brought within allowable limits of lead concentration, and for retailers required by § 80.22(b) of the regulations to make unleaded gasoline available, unleaded gasoline must be made available for sale within 6 days after the notification of the contamination; and (iii) that the retailer or his employee or agent will be notified in writing as to the procedure which will be used to remove the notice of contamination.

(e) The EPA inspector or the retailer, as directed by the Regional Administrator or his delegate, will post a notice of contamination on each pump dispensing the contaminated gasoline, in a prominent position, indicating that the gasoline is contaminated with lead or phosphorus and that further sale of gasoline from the pump is prohibited until the contaminated gasoline is disposed of or made to comply with federal specifications, and the notice is removed.

(f) The Fuel Inspector will use the second sample of gasoline for confirmatory laboratory testing.

Disposition or cure of the gasoline will be allowed in any reasonable manner, but not by sale or otherwise dispensing it from a pump which is subject to a notice of contamination. The retailer, for instance, may transfer the contaminated gasoline (but not by dispensing from a pump subject to a notice of contamination) into storage tanks containing leaded gasoline and sell the gasoline through pumps labeled for sale of leaded gasoline, or he may introduce sufficient quantities of unleaded gasoline into the storage tank containing the contaminated gasoline so as to dilute it until the mixture is in compliance with regulations. Alternatively, the retailer may return the contaminated gasoline to his supplier and replace it with unleaded. The retailer will in most cases be automatically released from the prohibition of the regulation upon the mailing to the Regional Administrator or his delegate of a notarized statement indicating the method of disposition or cure of the contaminated gasoline. However, the Regional Administrator or his authorized representative may inform the recipient of the notification that he will require confirmation by specific procedures provided for in the regulations that gasoline is uncontaminated before the release of the prohibition is authorized. He may, further, provide procedures for the retailer to obtain extensions of the 6-day time period if circumstances beyond the control of the retailer make compliance impossible.

An additional alternative available to the retailer to obtain release from the prohibition is for him to have the atomic absorption laboratory test for lead (or the laboratory test for phosphorus, as applicable) performed on the gasoline subject to the notice by the procedures prescribed in § 80.3, Appendix B (or § 80.3, Appendix A, as applicable). If the results from the test indicate that the sample contains 0.05 or less gram of lead per gallon (or 0.005 or less gram of phosphorus, as applicable), and this is uncontradicted by laboratory test results obtained by the Agency, which have been made known to the retailer, the retailer will be released from the prohibition upon submission of a notarized statement of satisfactory test results to the appropriate personnel at the EPA regional office. Documentation of the test results and that the proper procedures were followed may be required by the regional office.

The procedures outlined by the proposed regulations are necessary both to reduce the possibility of catalyst control system deactivation and to reestablish the availability of unleaded gasoline as quickly as practicable. In order to ensure that the proposed requirements are fully complied with, the proposed regulations explicitly identify actions on the part of the retailer which will constitute violations and will render him liable to the civil penalty provision provided for the § 80.5. These violations are in addition to those already existing under § 80.22. Specifically these actions are: (i) The sale, offering for sale or dispensing of contaminated gasoline from a pump subject to a notice of contamination after the retailer has been notified of the contamination and prior to the disposition or cure of the contaminated gasoline and removal of the notice of contamination; (ii) the removal of a notice of contamination prior to the time that the conditions for removal of such notice have been complied with; and, (iii) the filing with the Regional Administrator or his delegate of false or misleading information concerning a material fact in regard to the release of the prohibition on sale of the contaminated gasoline. A delay in making unleaded gasoline available, if no extension of the time period has been granted, is also a violation, but it covered under § 80.22(b).

Interested persons may submit written comments on the proposed test and regulatory provisions in triplicate to the Director, Mobile Source Enforcement Division (EG–340), Environmental Protection Agency, 401 M Street, SW., Washington, D.C. 20460. All relevant comments postmarked on or before December 12, 1974, will be considered. Comments received will be available for public inspection during normal working hours (8:00 a.m. to 4:30 p.m.) at the Freedom of Information Center, Waterside Mall, 401 M Street, SW., Washington, D.C. 20460.

This notice of proposed rulemaking is issued under the authority of sections 211 and 301 of the Clean Air Act, as amended (42 U.S.C., 1857f–6(c), 1857g (a)).

Dated: November 1, 1974.

JOHN QUARLES,
Acting Administrator.

It is proposed to amend Part 80 of Chapter I, Title 40 of the Code of Federal Regulations as follows:

1. In § 80.2, Paragraph (o) is added as follows:

§ 80.2 Definitions.

* * * * *

(o) "Contaminated Gasoline" means leaded gasoline as defined by paragraph (f) of this section which is sold, offered for sale, or dispensed from a pump labeled for the sale of unleaded gasoline in accordance with § 80.22(e)(1) or which is otherwise represented to be unleaded gasoline.

2. Section 80.3 is revised as follows:

§ 80.3 Test methods.

(a) The lead and phosphorus content of gasoline shall be determined in accordance with test methods set forth in the Appendices to this part.

(1) The laboratory test for the determination of phosphorus content in gasoline is described in Appendix A to this part and is essentially identical to the ASTM "Method of Test for the Determination of Phosphorus in Gasoline," #D3221–73 and shall be hereafter referred to as "Lab Test for Phosphorus" (LTP).

(2) The laboratory test for the determination of lead content in gasoline is described in Appendix B and is essentially identical to the ASTM "Standard Method of Test for Lead in Gasoline by Atomic Absorption Spectrometry," #D3237–73 and shall be hereafter referred to as "Lab Test for Lead" (LTL).

(3) The field test for the determination of lead content in gasoline shall be the "Field Test for Lead" (FTL) set forth in Appendix C of this part.

§ 80.22 [Amended]

(3) Renumber § 80.22(a) as § 80.22 (a)(1).

(4) Establish new § 80.22(a)(2).

(2) Any of the following actions on the part of a retailer or his employee or

agent shall constitute a violation of this subpart and subject the retailer to penalties as set forth in § 80.5.

(i) Sale, offering for sale, or dispensing gasoline from a pump after the retailer or his employee or agent has been notified pursuant to § 80.30(b) or § 80.30 (c) that gasoline in a storage tank which serves that pump is contaminated gasoline and prior to removal of a notice of contamination in accordance with § 80.32.

(ii) Removal or defacement of a notice of contamination from a pump prior to the time that each of the conditions of § 80.32 has been fully complied with.

(iii) Filing of false or misleading information with regard to a material fact in any submission to EPA regarding disposition or cure of contaminated gasoline, or the results of any test on gasoline.

(5) The first sentence of § 80.23 is revised to refer to paragraph (a)(1) of § 80.22.

(6) A new Subpart C is added as follows:

Subpart C—Notice of Contamination

Sec.
80.30 Testing: Notice of contamination.
80.31 Disposition or cure of contaminated gasoline.
80.32 Removal of notice of contamination.

AUTHORITY: Secs. 211, 301(a), Clean Air Act, as amended, (42 U.S.C. 1857f-6(c), 1857g(a)).

Subpart C—Notice of Contamination

§ 80.30 Testing: Notice of contamination.

(a) The Regional Administrator or his authorized representative may test gasoline samples from pumps dispensing gasoline represented as unleaded.

(b) If the result of a FTL, LTL, or LTP indicates that gasoline is contaminated, the retailer or his employee or agent will be so notified in writing.

(c) A notice will be affixed by an EPA inspector or the retailer as directed by the Regional Administrator or his authorized representative to each pump serviced by the underground tank servicing the pump from which the sample of contaminated gasoline was taken, in a prominent and conspicuous location, stating: "The gasoline in this pump has been found contaminated by excess lead or phosphorus additives. Federal law prohibits sale of gasoline from this pump until the contamination is eliminated, and this notice is removed in accordance with EPA regulations (40 CFR 80.32)." *Provided, however,* That the notice need not be affixed if the retailer notifies the Regional Administrator or his authorized representative that the gasoline represented by the sample found to be contaminated has been sold or disposed of and replaced by unleaded gasoline in conformity with the requirements of this part.

§ 80.31 Disposition or cure of contaminated gasoline.

(a) Disposition or cure of contaminated gasoline shall be made in any reasonable manner, but may not be accomplished by sale as unleaded gasoline or as leaded gasoline from any pump labeled for the sale of unleaded gasoline or from a pump subject to a notice of contamination.

(b) (1) Disposition or cure of contaminated gasoline shall be accomplished within 6 days following issuance of the notice of contamination. The retailer or his employee or agent shall notify the Regional Administrator or his authorized representative by mailing information detailing the method of disposition or cure of the gasoline or mailing results of an LTL or LTP, as applicable, showing that the gasoline is not, in fact, contaminated. This information shall be contained in a notarized statement.

(2) Any retailer required by § 80.22(b) to make unleaded gasoline available shall make unleaded gasoline available within 6 days following issuance of the notice of contamination.

(3) Extension of the 6 day time period may be granted by the Regional Administrator or his authorized representative upon receipt of an application for extension from the retailer demonstrating to the satisfaction of the Regional Administrator why he cannot comply with such time period. Such application may be made verbally.

§ 80.32 Removal of notice of contamination.

(a) The provision of § 80.22(a)(2) (i) and (ii) shall no longer apply upon satisfaction of the following conditions:

(1) Disposition or cure of the contaminated gasoline has been completed or results of an LTL or LTP, as applicable, obtained by the retailer or his employee or agent demonstrate that the gasoline in question is not contaminated. If EPA has tested this gasoline by an LTL or LTP, as applicable and has determined that the gasoline is contaminated and has so notified the retailer or his employee or agent, the notice of contamination may be removed only by disposition or cure of the contaminated gasoline;

(2) A notarized statement containing information regarding disposition, cure or satisfactory test results has been sent to the Regional Administrator or his authorized representative; and

(3) Fulfillment of the following additional requirements, if any, which the Regional Administrator or his authorized representative has made known to the notice recipient at the time of notification, to provide confirmation that the cured or replacement gasoline is uncontaminated:

(i) Submission of a sample of the cured or replacement unleaded gasoline,

(ii) Submission of the results of an LTL or LTP, as applicable, obtained by the retailer or his employee or agent of a sample of the cured or replacement unleaded gasoline,

(iii) Submission of a notarized statement signed by a distributor, his employee, or agent that unleaded gasoline was delivered to cure or replace the contaminated gasoline at the retail outlet. The statement shall contain such additional information with regard to the delivered gasoline as the Regional Administrator or his authorized representative may require. These requirements will be considered fulfilled when the sample, test result, and/or statement have been sent to the Regional Administrator or his authorized representative.

7. A new Appendix C is added as follows:

FIELD TEST FOR LEAD (FTL)

APPENDIX C—FIELD TEST FOR DETERMINATION OF LEAD CONTENT IN UNLEADED GASOLINE

1. *Scope.*

1.1 This method is intended for use in the field by nontechnical people for the quantitative measurement of lead in unleaded gasoline in the range from 0.01 to 0.10 g Pb/U.S. gal. The method applies to all commercial gasolines and responds to all types of lead alkyls as well as to other organic and inorganic forms of lead.

2. *Summary of Method.*

2.1 The gasoline is treated with iodine and tetraethyl ammonium chloride in chloroform and subjected to ultraviolet light. The lead alkyls form water-soluble lead alkyl iodides, which are removed from the gasoline by shaking it with an aqueous ammonium nitrate solution. The aqueous extract is filtered into a solution of 4-(2-pyridylazo)-resorcinol disodium salt (PAR) and ammonium hydroxide. The lead is determined by measuring its PAR complex colorimetrically at 490 nm using a previously prepared calibration curve.

3. *Apparatus.*

3.1 "Ultraviolet Lamp," long wavelength, 3660 Å, placed in a standard 4-watt fluorescent fixture.

3.2 Measuring Block, aluminum, drilled to hold an 18 by 150-mm test tube, with a mark at a level equal to 5.0 ml of liquid in the test tube.

3.3 "Colorimeter, portable," capable of operating at 490 mm. Any equivalent instrument capable of measurement near 514 mm (the optimum Pb-PAR complex wavelength) may be used.

3.4 "Test tubes" borosilicate, 18 by 150 mm.

3.5 "Pipets," glass, dropping, capable of delivering 2.0 ml with a 2-ml bulb.

NOTE 1: Caution. Gasoline or any of the reagents must not come in contact with rubber. If this happens, discard the bulb and pipet and start again.

3.6 "Funnel," plastic, 2.5 inch inside diameter.

3.7 "Filter paper," ashless, 11.00 cm in diameter.

3.8 "Graduated cylinder," plastic, 10-ml.

3.9 Glass Vials, with caps, disposable, 1-oz. capacity.

4. *Reagents.*

4.1 *Purity of reagents.* Reagent grade chemical shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 Ammonium Hydroxide (sp. gr. 0.90)—Concentrated ammonium hydroxide (NH_4OH).

4.3 Ammonium Nitrate Solution (Reagent B)—Dissolve 15.0±0.1 g of ammonium

nitrate (NH_4NO_3) in 750 ml of water in a 1-litre volumetric flash. Dilute to the mark with water.

4.4 *Chloroform ($CHCl_3$).*

4.5 *Disodium Salt of 4-(2-pyridylazo) Resorcinol Dihydrate (PAR, $2H_2O$) (Reagent C).* Dissolve 25.0±0.1 mg of PAR in 750 ml of water in a 1 liter volumetric flask. Add 10.0±0.1 ml concentrated NH_4OH. Dilute to the mark with water. Store this in brown bottles out of direct sunlight or in the dark.

NOTE 2: Caution—Low results are obtained if the mono-sodium or unsalted PAR is used in this test.

4.6 *Gasoline, Lead-Free.* Gasoline containing less than 0.001 g PB/gal.

4.7 *Iodine.*

4.8 *Iodine/TEAC/$CHCl_3$ Solution (Reagent A).* Dissolve 1.000 g±1 mg of iodine in 75 ml of chloroform ($CHCl_3$) in a 100-ml volumetric flask. Add 1.000 g±1 mg of tetraethylammonium chloride (TEAC) and mix until dissolved. Dilute to the mark with $CHCl_3$.

NOTE 3: Solutions 4.3, 4.5, and 4.8 have been found to be stable for at least 2 months.

4.9 *Lead Standards.* This method was developed using lead standards prepared by addition of known amounts of various lead alkyls to be blended unleaded gasoline to cover the range of this method.

4.11 *Tetraethylammonium Chloride Monohydrate (TEAC).*

5. *Calibration.*

5.1 Prepare a calibration curve to be used with the portable colorimeter. Such a curve should be prepared as frequently as necessary to assure that accurate test results are being obtained. Use at least four gasoline standards of known lead content that cover the range from 0.01 to 0.10 g Pb/gal. to prepare the curve.

5.1.1 Rinse the 2-ml graduated pipet three times with the gasoline sample. Add 2.0 ml of the sample to a 1-oz glass vial. Add 2.0 ml of iodine/TEAC/$CHCl_3$ solution (Reagent A) from another pipet, to the vial containing the gasoline. Tightly cap the vial.

5.1.2 Place the vial on the ultraviolet light and set the timer to give the sample a 3-minute exposure. *Caution.* Ultraviolet light can be harmful to the eyes. Do not remove any protective shields. DO NOT stare at the light.

5.1.3 After exposure, remove and uncap the vial. Measure 10.0 ml of ammonium nitrate solution (Reagent B) into the 10-ml graduated cylinder. Add this to the vial containing the sample. Recap and shake the vial vigorously for 1 minute.

5.1.4 Place a clean 18-mm test tube in the aluminum measuring block. Add 5.0 ml of PAR solution (Reagent C) to the test tube using the mark on the block such that the upper level of liquid in the tube is equal to the mark on the block. Place the plastic funnel in the test tube. Fold a piece of filter paper and place in the funnel.

5.1.5 When the two layers of liquid in the vial have separated (5.1.3), pour the entire contents of the vial inside the filter paper. The aqueous layer will remain in the filter paper. Tap the funnel to add any remaining drops of aqueous solution to the test tube. Remove the funnel and discard its contents. Swirl the test tube gently using a wrist action to obtain a uniform color.

NOTE 4: The lead-PAR complex formed in 5.1.5 must be measured within 10 minutes after starting 5.1.5.

NOTE 5: A few drops of the organic layer may come through the filter paper. This will not alter the results and can therefore be tolerated. However, if more than 10 drops do come through, refilter the aqueous layer through a fresh filter paper into a clean empty test tube.

NOTE 6: Swirling may cause air bubbles to be trapped in the liquid. Wait for these to settle before continuing. Wipe the test tube off with a clean towel to remove any fingerprints that may be present on the surface of the tube.

5.2 Zero and standardize the colorimeter as follows:

5.2.1 Set the colorimeter at 490 nm. Set zero absorbance (100 percent transmittance) with water in an 18-mm test tube. Read and record the absorbance (or percent transmittance) obtained for the standards prepared under 5.1.5.

5.2.2 Plot the absorbance values versus concentration on rectangular coordinate paper. (If percent transmittance values are used, plot them versus concentration using semilog paper, with the percent transmittance value on the log scale.) Draw a best fit line by eye. When plotting absorbance versus concentration, note that the curve does not pass through the origin.

6. *Procedure.*

6.1 Prepare the sample in accordance with the directions given in 5.1.1 through 5.1.5.

6.2 Place the test tube containing the water in the colorimeter and set the absorbance to zero, or the transmittance to 100%.

6.3 Place the sample in the colorimeter and read the absorbance or percent transmittance.

NOTE 7: *Time limit.* The PAR-lead solution obtained for the sample in 6.1 must be read within 10 minutes after the step described in 5.1.5.

6.4 From the calibration curve, find the lead content of the sample. Determine the lead content to the nearest 0.001 g/gal value.

6.4.1 Round the value obtained in 6.4 to the nearest 0.01 g/gal value. This will be the value reported.

7. *Precision.*

7.1 The following criteria should be used for judging the acceptability of results (95 percent confidence):

7.1.1 *Repeatability.* Duplicate results by the same operator should be considered suspect if they differ by more than 0.01 g/gal.

7.1.2 *Reproducibility.* The results submitted by each of two laboratories should not be considered suspect unless the results differ by more than 0.02 g/gal.

APPENDIX D—[REDESIGNATED]

2. Appendix C—List of counties with population densities less than 50 persons per square mile (excluding cities with populations equal to or greater than 50,000 persons) is changed to Appendix D.

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